



DATA VALIDATION REPORT

Gold King Mine Release Incident

SAMPLE DELIVERY GROUP: 280-74118-1

Prepared by

MEC^X
12269 East Vassar Drive
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I. INTRODUCTION

Task Order Title: Gold King Mine Release Incident
Project No.: 20408.012.001.0274.00
20408.012.001.0267.00
Sample Delivery Group: 280-74118-1
EPA Project Manager: Steve Way
Weston Project Manager: Dave Robinson
TDD No.: 0001/1508-04
Matrix: Sediment
QC Level: Stage 2A
No. of Samples: 4
No. of Reanalyses/Dilutions: 0
Laboratory: TestAmerica - Denver

Table 1. Sample Identification

<i>Location ID</i>	<i>Lab Sample Name</i>	<i>Matrix Type</i>	<i>Collection Date</i>	<i>Method</i>
GKMXRF018_082515	280-74118-3	Sediment	8/25/15 11:30 AM	6010C, 6020A, 7471A
GKMXRF019_082515	280-74118-4	Sediment	8/25/15 11:25 AM	6010C, 6020A, 7471A
GKMXRF021_082515	280-74118-5	Sediment	8/25/15 12:20 PM	6010C, 6020A, 7471A
GKMXRF025_082515	280-74118-6	Sediment	8/25/15 11:00 AM	6010C, 6020A, 7471A

II. Sample Management

Anomalies regarding sample management are listed below. The samples were received below the temperature limits of 4°C \pm 2°C, at 1.0°C. As the samples were not noted to be frozen or damaged, no qualifications were required. The samples were received intact and on ice. The chain-of-custody (COC) was appropriately signed and dated by field and laboratory personnel. The presence or absence of custody seals on the cooler was not specifically noted.

The following issues were noted:

- Hardness was requested on the COC; however, per previous instruction, the laboratory did not provide the results of this calculation.
- The COCs did not list CLP sample IDs, and none were provided. The laboratory logged the samples per the location IDs on the COCs.
- The presence or absence of sample tags was not noted in the case narrative, and sample tags were not listed on the COCs.

**Data Qualifier Reference Table**

Qualifier	Organics	Inorganics
U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit. The associated value is the quantitation limit or the estimated detection limit for dioxins or PCB congeners.	The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit. The associated value is the sample detection limit or the quantitation limit for perchlorate only.
UB	The analyte was detected in the sample and in either the associated laboratory blank or field blank. If detected below the reporting limit (RL) the analyte result was reported as non-detected at the RL due to blank contamination. If detected above the RL, the analyte result was reported as non-detected at the reported result due to blank contamination.	The analyte was detected in the sample and in either the associated laboratory blank or field blank. If detected below the reporting limit (RL) the analyte result was reported as non-detected at the RL due to blank contamination. If detected above the RL, the analyte result was reported as non-detected at the reported result due to blank contamination.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
J+	Not applicable	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample, and may have a potential positive bias.
J-	Not applicable	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample, and may have a potential negative bias.



Qualifier	Organics	Inorganics
UJ	The analyte was not deemed above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.	The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.
UJB	The analyte was detected in the sample and in either the associated laboratory blank or field blank; the analyte result was reported as non-detected at either the RL or the reported result. The reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.	The analyte was detected in the sample and in either the associated laboratory blank or field blank; the analyte result was reported as non-detected at either the RL or the reported result. The reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."	Not applicable.
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.	Not applicable.
R	The data are unusable. The sample results are rejected due to serious deficiencies in the ability to analyze the sample and to meet quality control criteria. The presence or absence of the analyte cannot be verified.	The data are unusable. The sample results are rejected due to serious deficiencies in the ability to analyze the sample and to meet quality control criteria. The presence or absence of the analyte cannot be verified.

**Qualification Code Reference Table**

Qualifier	Organics	Inorganics
H	Holding times were exceeded.	Holding times were exceeded.
S	Surrogate recovery was outside QC limits.	The sequence or number of standards used for the calibration was incorrect
C	Calibration %RSD or %D was noncompliant.	Correlation coefficient is <0.995 or calibration was noncompliant.
R	Calibration RRF was <0.05.	%R for calibration is not within control limits.
B	Presumed contamination as indicated by the preparation (method) blank results.	Presumed contamination as indicated by the preparation (method) or calibration blank results.
L	Laboratory Blank Spike/Blank Spike Duplicate %R was not within control limits.	Laboratory Control Sample %R was not within control limits.
L1	LCS/LCSD RPD was outside control limits.	LCS/LCSD RPD was outside control limits.
Q	MS/MSD recovery was poor.	MS recovery was poor.
Q1	MS/MSD RPD was outside control limits.	MS/MSD RPD was outside control limits.
E	Not applicable.	Duplicates showed poor agreement.
I	Internal standard performance was unsatisfactory.	ICP ICS results were unsatisfactory.
A	Not applicable.	ICP Serial Dilution %D were not within control limits.
M	Tuning (BFB or DFTPP) was noncompliant.	ICPMS tune was not compliant.
T	Presumed contamination as indicated by the trip blank results.	Not applicable.
+	False positive – reported compound was not present.	Not applicable.
-	False negative – compound was present but not reported.	Not applicable.
F	Presumed contamination as indicated by the FB or ER results.	Presumed contamination as indicated by the FB or ER results.
F1	Field duplicate results were outside the control limit.	Field duplicate results were outside the control limit.
\$	Reported result or other information was incorrect.	Reported result or other information was incorrect.



Qualifier	Organics	Inorganics
?	TIC identity or reported retention time has been changed.	Not applicable.
D	The analysis with this flag should not be used because another more technically sound analysis is available.	The analysis with this flag should not be used because another more technically sound analysis is available.
P	Instrument performance for pesticides was poor.	Post Digestion Spike recovery was not within control limits.
*II, *III	Unusual problems found with the data that have been described in Section II, "Sample Management," or Section III, "Method Analyses." The number following the asterisk (*) will indicate the report section where a description of the problem can be found.	Unusual problems found with the data that have been described in Section II, "Sample Management," or Section III, "Method Analyses." The number following the asterisk (*) will indicate the report section where a description of the problem can be found.



III. Method Analyses

A. Contract Laboratory Program Statement of Work for Inorganic Superfund Methods, 6010C, 6020A, 7471A — Metals and Mercury

Reviewed By: P. Meeks

Date Reviewed: September 26, 2015

The samples listed in Table 1 for these analyses were validated based on the guidelines outlined in the *Sampling and Analysis Plan/Quality Assurance Project Plan for Gold King Mine Release, Silverton, San Juan County, Colorado* (2015), *United States Environmental Protection Agency Contract Laboratory Program Statement of Work for Inorganic Superfund Methods, EPA Methods 6010C, 6020A, 7471A*, and the *National Functional Guidelines for Inorganic Superfund Data Review* (2010).

- Holding Times: The analytical holding times, 28 days for mercury and six months for the remaining metals, was met.
- Analytical Method Blanks: There were detects reported in the method blanks, but none were sufficient to qualify the site samples.
- Laboratory Control Samples (LCS): The recoveries were within laboratory control limits of 87-111% for mercury and the method control limits of 80-120% for the remaining analytes.
- Laboratory Duplicates: Laboratory duplicate analyses were performed on samples GKMXRf018_082515 the 6010C analytes, on GKMXRf019_082515 for the 6020A analytes, and on GKMXRf021_082515 for mercury. The relative percent differences (RPDs) for antimony (53%), cadmium (87%), molybdenum (143%), and zinc (66%) exceeded the control limit; therefore, results for these analytes were qualified as estimated (J) in the samples. The remaining RPDs were $\leq 20\%$ for results greater than $5\times$ the reporting limit (RL) and within $\pm RL$ for results less than $5\times$ the RL.
- Matrix Spike/Matrix Spike Duplicate (MS/MSD): MS/MSD analyses were performed on samples GKMXRf018_082515, on GKMXRf019_082515 for the 6020A analytes, and on GKMXRf021_082515 for mercury. Results were not assessed when the native concentration was more than $4\times$ the spike amount. Potassium (128%/128%), antimony (23%/17%), molybdenum (137%/ acceptable), arsenic (acceptable/33%), and barium (acceptable/54%) were recovered outside the control limits. Results associated with low recoveries were qualified as estimated with a potential low bias (J-) and results associated with high recoveries were qualified as estimated with a potential high bias (J+). The remaining recoveries were within the laboratory control limits of 87-111% for mercury and the method control limits of 75-125% for the remaining analytes.



RPDs for antimony (24%), arsenic (26%), and molybdenum (38%) exceeded the control limit; therefore, the results (all detects) were qualified as estimated (J). The remaining RPDs were $\leq 20\%$.

- Post Digestion Spike (PDS): No PDS analyses were performed on a sample in this SDG.
- Serial Dilution: There were no serial dilution analyses performed in this SDG.
- Field QC Samples: Field QC samples were evaluated, and if necessary, qualified based on method blanks and other laboratory QC results affecting the usability of the field QC data. Any remaining detects were used to evaluate the associated site samples. Following are findings associated with field QC samples:
 - Field Blanks and Equipment Rinsates: No field blank or equipment rinsate samples were identified in this SDG.
 - Field Duplicates: There were no field duplicate samples identified in this SDG.

Validated Sample Result Forms: 280-74118-1

Analysis Method 6010C

Sample Name GKMXRf018_082515

Matrix Type: Solid

Lab Sample Name: 280-74118-3

Sample Date: 8/25/2015 11:30:00 AM

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
Aluminum	T	7429-90-5	7400	9	1.4	mg/Kg			
Calcium	T	7440-70-2	1200	45	13	mg/Kg	B		
Iron	T	7439-89-6	47000	13	3.4	mg/Kg	B		
Magnesium	T	7439-95-4	3800	18	3.3	mg/Kg	B		
Potassium	T	7440-09-7	1300	270	37	mg/Kg		J+	Q
Sodium	T	7440-23-5	120	450	53	mg/Kg	J	J	

Sample Name GKMXRf019_082515

Matrix Type: Solid

Lab Sample Name: 280-74118-4

Sample Date: 8/25/2015 11:25:00 AM

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
Aluminum	T	7429-90-5	13000	9.5	1.5	mg/Kg			
Calcium	T	7440-70-2	2000	48	13	mg/Kg	B		
Iron	T	7439-89-6	67000	14	3.6	mg/Kg	B		
Magnesium	T	7439-95-4	7700	19	3.5	mg/Kg	B		
Potassium	T	7440-09-7	890	290	39	mg/Kg		J+	Q
Sodium	T	7440-23-5	56	480	56	mg/Kg		U	

Sample Name GKMXRf021_082515

Matrix Type: Solid

Lab Sample Name: 280-74118-5

Sample Date: 8/25/2015 12:20:00 PM

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
Aluminum	T	7429-90-5	9500	9.7	1.5	mg/Kg			
Calcium	T	7440-70-2	660	48	14	mg/Kg	B		
Iron	T	7439-89-6	31000	15	3.7	mg/Kg	B		
Magnesium	T	7439-95-4	5600	19	3.6	mg/Kg	B		
Potassium	T	7440-09-7	950	290	40	mg/Kg		J+	Q
Sodium	T	7440-23-5	93	480	57	mg/Kg	J	J	

Sample Name GKMXRf025_082515

Matrix Type: Solid

Lab Sample Name: 280-74118-6

Sample Date: 8/25/2015 11:00:00 AM

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
Aluminum	T	7429-90-5	6100	9.1	1.4	mg/Kg			
Calcium	T	7440-70-2	1000	45	13	mg/Kg	B		

Analysis Method 6010C

Iron	T	7439-89-6	45000	14	3.4	mg/Kg	B		
Magnesium	T	7439-95-4	3300	18	3.4	mg/Kg	B		
Potassium	T	7440-09-7	1200	270	37	mg/Kg		J+	Q
Sodium	T	7440-23-5	91	450	54	mg/Kg	J	J	

Analysis Method 6020A

Sample Name	GKMXRF018_082515					Matrix Type:	Solid
Lab Sample Name:	280-74118-3		Sample Date:	8/25/2015 11:30:00 AM			

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
Antimony	T	7440-36-0	590	200	14	ug/Kg		J-	E, Q, Q1
Arsenic	T	7440-38-2	27000	590	50	ug/Kg		J-	Q, Q1
Barium	T	7440-39-3	51000	200	70	ug/Kg	B	J+	Q
Beryllium	T	7440-41-7	450	99	22	ug/Kg			
Cadmium	T	7440-43-9	1100	99	9.3	ug/Kg		J	E
Chromium	T	7440-47-3	5100	200	75	ug/Kg			
Cobalt	T	7440-48-4	4700	99	6.5	ug/Kg			
Copper	T	7440-50-8	110000	250	70	ug/Kg	B ^		
Lead	T	7439-92-1	540000	1500	180	ug/Kg			
Manganese	T	7439-96-5	530000	2500	330	ug/Kg	B		
Molybdenum	T	7439-98-7	7100	200	17	ug/Kg		J+	E, Q, Q1
Nickel	T	7440-02-0	4100	150	25	ug/Kg			
Selenium	T	7782-49-2	2200	490	130	ug/Kg			
Silver	T	7440-22-4	2800	99	20	ug/Kg			
Thallium	T	7440-28-0	260	99	3.5	ug/Kg	B		
Vanadium	T	7440-62-2	25000	490	38	ug/Kg			
Zinc	T	7440-66-6	290000	990	310	ug/Kg		J	E

Sample Name	GKMXRF019_082515					Matrix Type:	Solid
Lab Sample Name:	280-74118-4		Sample Date:	8/25/2015 11:25:00 AM			

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
Antimony	T	7440-36-0	750	190	14	ug/Kg	F1 F2	J-	E, Q, Q1
Arsenic	T	7440-38-2	33000	580	49	ug/Kg	F1 F2	J-	Q, Q1
Barium	T	7440-39-3	66000	190	68	ug/Kg	B F1	J+	Q
Beryllium	T	7440-41-7	560	97	22	ug/Kg			
Cadmium	T	7440-43-9	1900	97	9.1	ug/Kg		J	E
Chromium	T	7440-47-3	6200	190	74	ug/Kg			
Cobalt	T	7440-48-4	10000	97	6.4	ug/Kg			
Copper	T	7440-50-8	110000	240	69	ug/Kg	B ^		
Lead	T	7439-92-1	480000	7300	880	ug/Kg			
Manganese	T	7439-96-5	1700000	12000	1600	ug/Kg	B		
Molybdenum	T	7439-98-7	4200	190	17	ug/Kg	F1 F2	J+	E, Q, Q1

Analysis Method 6020A

Nickel	T	7440-02-0	5500	150	25	ug/Kg			
Selenium	T	7782-49-2	880	480	130	ug/Kg			
Silver	T	7440-22-4	1600	97	20	ug/Kg			
Thallium	T	7440-28-0	250	97	3.4	ug/Kg	B		
Vanadium	T	7440-62-2	29000	480	37	ug/Kg	F1		
Zinc	T	7440-66-6	570000	48000	15000	ug/Kg	F2	J	E

Sample Name GKMXR021_082515

Matrix Type: Solid

Lab Sample Name: 280-74118-5

Sample Date: 8/25/2015 12:20:00 PM

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
Antimony	T	7440-36-0	160	190	13	ug/Kg	J	J-	E, Q, Q1
Arsenic	T	7440-38-2	25000	580	49	ug/Kg		J-	Q, Q1
Barium	T	7440-39-3	100000	190	68	ug/Kg	B	J+	Q
Beryllium	T	7440-41-7	350	96	22	ug/Kg			
Cadmium	T	7440-43-9	260	96	9	ug/Kg		J	E
Chromium	T	7440-47-3	7200	190	73	ug/Kg			
Cobalt	T	7440-48-4	4500	96	6.4	ug/Kg			
Copper	T	7440-50-8	58000	240	68	ug/Kg	B ^		
Lead	T	7439-92-1	410000	140	17	ug/Kg			
Manganese	T	7439-96-5	1100000	240	32	ug/Kg	B ^		
Molybdenum	T	7439-98-7	2200	190	17	ug/Kg		J+	E, Q, Q1
Nickel	T	7440-02-0	3700	140	24	ug/Kg			
Selenium	T	7782-49-2	960	480	130	ug/Kg			
Silver	T	7440-22-4	1400	96	19	ug/Kg			
Thallium	T	7440-28-0	190	96	3.4	ug/Kg	B		
Vanadium	T	7440-62-2	29000	480	37	ug/Kg			
Zinc	T	7440-66-6	140000	960	300	ug/Kg		J	E

Sample Name GKMXR025_082515

Matrix Type: Solid

Lab Sample Name: 280-74118-6

Sample Date: 8/25/2015 11:00:00 AM

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
Antimony	T	7440-36-0	520	190	14	ug/Kg		J-	E, Q, Q1
Arsenic	T	7440-38-2	27000	580	49	ug/Kg		J-	Q, Q1
Barium	T	7440-39-3	54000	190	68	ug/Kg	B	J+	Q
Beryllium	T	7440-41-7	320	97	22	ug/Kg			
Cadmium	T	7440-43-9	830	97	9.1	ug/Kg		J	E
Chromium	T	7440-47-3	4800	190	74	ug/Kg			
Cobalt	T	7440-48-4	3400	97	6.4	ug/Kg			
Copper	T	7440-50-8	120000	240	69	ug/Kg	B ^		
Lead	T	7439-92-1	430000	1500	180	ug/Kg			
Manganese	T	7439-96-5	520000	240	32	ug/Kg	B ^		

Analysis Method 6020A

Molybdenum	T	7439-98-7	9800	190	17	ug/Kg	J+	E, Q, Q1
Nickel	T	7440-02-0	3200	150	24	ug/Kg		
Selenium	T	7782-49-2	2000	480	130	ug/Kg		
Silver	T	7440-22-4	3000	97	20	ug/Kg		
Thallium	T	7440-28-0	270	97	3.4	ug/Kg	B	
Vanadium	T	7440-62-2	24000	480	37	ug/Kg		
Zinc	T	7440-66-6	260000	970	310	ug/Kg	J	E

Analysis Method 7471A

Sample Name GKMXRf018_082515 **Matrix Type:** Solid

Lab Sample Name: 280-74118-3 **Sample Date:** 8/25/2015 11:30:00 AM

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
Mercury	T	7439-97-6	38	20	6.6	ug/Kg			

Sample Name GKMXRf019_082515 **Matrix Type:** Solid

Lab Sample Name: 280-74118-4 **Sample Date:** 8/25/2015 11:25:00 AM

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
Mercury	T	7439-97-6	46	20	6.6	ug/Kg			

Sample Name GKMXRf021_082515 **Matrix Type:** Solid

Lab Sample Name: 280-74118-5 **Sample Date:** 8/25/2015 12:20:00 PM

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
Mercury	T	7439-97-6	6.1	19	6.1	ug/Kg		U	

Sample Name GKMXRf025_082515 **Matrix Type:** Solid

Lab Sample Name: 280-74118-6 **Sample Date:** 8/25/2015 11:00:00 AM

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
Mercury	T	7439-97-6	41	20	6.5	ug/Kg			